

09/889901  
JCS9 Rec'd PCT/TC 24 JUL 2001

Practitioner's Docket No. MCA-400 PC/US

CHAPTER II

Preliminary Classification:

Proposed Class:

Subclass:

NOTE: "All applicants are requested to include a preliminary classification on newly filed patent applications. The preliminary classification, preferably class and subclass designations, should be identified in the upper right-hand corner of the letter of transmittal accompanying the application papers, for example 'Proposed Class 2, subclass 129.' " M.P.E.P. Section 601, 7th ed.

TRANSMITTAL LETTER  
TO THE UNITED STATES ELECTED OFFICE (EO/US)

(ENTRY INTO U.S. NATIONAL PHASE UNDER CHAPTER II)

PCT/US00/02193	27 January 2000	60/117,856	29 January 1999
INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY	DATE CLAIMED

METHOD OF FORMING A PERFLUORINATED, THERMOPLASTIC HOLLOW FIBER MODULE  
TITLE OF INVENTION

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APPLICANT(S)

Box PCT  
Commissioner for Patents  
Washington D.C. 20231  
ATTENTION: EO/US

CERTIFICATION UNDER 37 C.F.R. SECTION 1.10\*

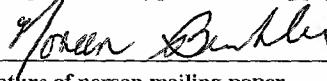
(Express Mail label number is **mandatory**.)

(Express Mail certification is **optional**.)

I hereby certify that this correspondence and the documents referred to as attached therein are being deposited with the United States Postal Service on this date July 24, 2001, in an envelope as "Express Mail Post Office to Addressee," Mailing Label Number ET683704046US, addressed to the: Assistant Commissioner for Patents, Washington, D.C. 20231.

Noreen Buckley

(type or print name of person mailing paper)

  
Signature of person mailing paper

**WARNING:** Certificate of mailing (first class) or facsimile transmission procedures of 37 C.F.R. Section 1.8 cannot be used to obtain a date of mailing or transmission for this correspondence.

**\*WARNING:** Each paper or fee filed by "Express Mail" **must** have the number of the "Express Mail" mailing label placed thereon prior to mailing. 37 C.F.R. Section 1.10(b).

"Since the filing of correspondence under [Section] 1.10 without the Express Mail mailing label thereon is an oversight that can be avoided by the exercise of reasonable care, requests for waiver of this requirement will **not** be granted on petition.' Notice of Oct. 24, 1996, 60 Fed. Reg. 56,439, at 56,442.

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**NOTE:** To avoid abandonment of the application, the applicant shall furnish to the USPTO, not later than 20 months from the priority date: (1) a copy of the international application, unless it has been previously communicated by the International Bureau or unless it was originally filed in the USPTO; and (2) the basic national fee (see 37 C.F.R. Section 1.492(a)). The 30-month time limit may not be extended. 37 C.F.R. Section 1.495.

**WARNING:** Where the items are those which can be submitted to complete the entry of the international application into the national phase are subsequent to 30 months from the priority date the application is still considered to be in the international state and if mailing procedures are utilized to obtain a date the express mail procedure of 37 C.F.R. Section 1.10 must be used (since international application papers are not covered by an ordinary certificate of mailing - See 37 C.F.R. Section 1.8).

**NOTE:** Documents and fees must be clearly identified as a submission to enter the national state under 35 USC 371 otherwise the submission will be considered as being made under 35 U.S.C. Section 111. 37 C.F.R. Section 1.494(f).

1. Applicant herewith submits to the United States Elected Office (EO/US) the following items under 35 U.S.C. 371:
  - a. [X] This express request to immediately begin national examination procedures (35 U.S.C. Section 371(f)).
  - b. [X] The U.S. National Fee (35 U.S.C. Section 371(c)(1)) and other fees (37 C.F.R. Section 1.492) as indicated below:

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## 2.Fees

CLAIMS FEE	(1) FOR	(2) NUMBER FILED	(3) NUMBER EXTRA	(4) RATE	(5) CALCULATIONS
[ ]*	TOTAL CLAIMS	27- 20 =	7	x \$ 18.00 =	\$126.00
	INDEPENDENT CLAIMS	3- 3 =	0	x \$ 80.00 =	0
MULTIPLE DEPENDENT CLAIM(S) (if applicable) + \$270.00					
BASIC FEE**	<p>[ ] U.S. PTO WAS INTERNATIONAL PRELIMINARY EXAMINATION AUTHORITY            Where an International preliminary examination fee as set forth in Section 1.482 has been paid on the international application to the U.S. PTO:</p> <p>[ ] and the international preliminary examination report states that the criteria of novelty, inventive step (non-obviousness) and industrial activity, as defined in PCT Article 33(2) to (4) have been satisfied for all the claims presented in the application entering the national stage (37 C.F.R. Section 1.492(a)(4)) ..... \$100.00</p> <p>[ ] and the above requirements are not met (37 C.F.R. Section 1.492(a)(1)) ..... \$690.00</p> <p>[X] U.S. PTO WAS NOT INTERNATIONAL PRELIMINARY EXAMINATION AUTHORITY            Where no international preliminary examination fee as set forth in Section 1.482 has been paid to the U.S. PTO, and payment of an international search fee as set forth in Section 1.445(a)(2) to the U.S. PTO:</p> <p>[ ] has been paid (37 C.F.R. 1.492(a)(2)) ..... \$710.00</p> <p>[ ] has not been paid (37 C.F.R. 1.492(a)(3)) ..... \$1,000.00</p> <p>[X] where a search report on the international application has been prepared by the European Patent Office or the Japanese Patent Office (37 C.F.R. Section 1.492(a)(5)) ..... \$860.00</p>				
Total of above Calculations					=
Reduction by 1/2 for filing by small entity, if applicable. Affidavit must be filed. (note 37 C.F.R. Sections 1.9, 1.27, 1.28)					-
Subtotal					986.00
Total National Fee					\$986.00
Fee for recording the enclosed assignment document \$40.00 (37 C.F.R. 1.21(h)). (See Item 13 below). See attached "ASSIGNMENT COVER SHEET".					\$ 40.00
TOTAL					Total Fees enclosed \$1,026.00

\* See attached Preliminary Amendment Reducing the Number of Claims.

- i. [X] A check in the amount of \$1,026.00 to cover the above fees is enclosed.
- ii. [ ] Please charge Account No. \_\_\_\_\_ in the amount of \$ \_\_\_\_\_.  
 A duplicate copy of this sheet is enclosed.

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**\*\* WARNING:** "To avoid abandonment of the application the applicant shall furnish to the United States Patent and Trademark Office not later than the expiration of 30 months from the priority date: \* \* \* (2) the basic national fee (see Section 1.492(a)) The 30-month time limit may not be extended." 37 C.F.R. Section 1.495(b)

**WARNING:** If the translation of the international application and/or the oath or declaration have not been submitted by the applicant within thirty (30) months from the priority date, such requirements may be met within a time period set by the Office. 37 C.F.R. Section 1.495(b)(2). The payment of the surcharge set forth in Section 1.492(e) is required as a condition for accepting the oath or declaration later than thirty (30) months after the priority date. The payment of the processing fee set forth in Section 1.492(f) is required for acceptance of an English translation later than thirty (30) months after the priority date. Failure to comply with these requirements will result in abandonment of the application. The provisions of Section 1.136 apply to the period which is set. Notice of Jan. 3, 1993, 1147 O.G. 29 to 40.

3.  A copy of the International application as filed (35 U.S.C. Section 371(c)(2)):

**NOTE:** Section 1.495 (b) was amended to require that the basic national fee and a copy of the international application must be filed with the Office by 30 months from the priority date to avoid abandonment "The International Bureau normally provides the copy of the international application to the Office in accordance with PCT Article 20. At the same time, the International Bureau notifies applicant of the communication to the Office. In accordance with PCT Rule 47.1, that notice shall be accepted by all designated offices as conclusive evidence that the communication has duly taken place. Thus, if the applicant desires to enter the national stage, the applicant normally need only check to be sure the notice from the International Bureau has been received and then pay the basic national fee by 30 months from the priority date." Notice of Jan. 7, 1993, 1147 O.G. 29 to 40, at 35-36. See item 14c below.

- a.  is transmitted herewith.
- b.  is not required, as the application was filed with the United States Receiving Office.
- c.  has been transmitted
  - i.  by the International Bureau.  
Date of mailing of the application (from form PCT/IB/308): 03 August 2000.
  - ii.  by applicant on \_\_\_\_\_  
Date

4.  A translation of the International application into the English language (35 U.S.C. Section 371(c)(2)):

- a.  is transmitted herewith.
- b.  is not required as the application was filed in English.
- c.  was previously transmitted by applicant on \_\_\_\_\_  
Date
- d.  will follow.

5.  Amendments to the claims of the International application under PCT Article 19 (35 U.S.C. Section 371(c)(3)):

**NOTE:** The Notice of January 7, 1993 points out that 37 C.F.R. Section 1.495(a) was amended to clarify the existing and continuing practice that PCT Article 19 amendments must be submitted by 30 months from the priority date and this deadline may not be extended. The Notice further advises that: "The failure to do so will not result in loss of the subject matter of the PCT Article 19 amendments. Applicant may submit that subject matter in a preliminary amendment filed under Section 1.121. In many cases, filing an amendment under Section 1.121 is preferable since grammatical or idiomatic errors may be corrected." 1147 O.G. 29-40, at 36.

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a. [ ] are transmitted herewith.

b. [ ] have been transmitted

- i. [ ] by the International Bureau.  
Date of mailing of the amendment (from form PCT/IB/308): \_\_\_\_\_.
- ii. [ ] by applicant on \_\_\_\_\_  
Date

c. [X] have not been transmitted as

- i. [X] applicant chose not to make amendments under PCT Article 19.  
Date of mailing of Search Report (from form PCT/ISA/210): \_\_\_\_\_.
- ii. [ ] the time limit for the submission of amendments has not yet expired. The amendments or a statement that amendments have not been made will be transmitted before the expiration of the time limit under PCT Rule 46.1.

6. [X] A translation of the amendments to the claims under PCT Article 19 (38 U.S.C. Section 371(c)(3)):

- a. [ ] is transmitted herewith.
- b. [ ] is not required as the amendments were made in the English language.
- c. [X] has not been transmitted for reasons indicated at point 5(c) above.

7. [X] A copy of the international examination report (PCT/IPEA/409)

- [ ] is transmitted herewith.
- [ ] is not required as the application was filed with the United States Receiving Office.
- [X] is not enclosed as Applicant did not file Chapter I or II

8. [ ] Annex(es) to the international preliminary examination report

- a. [ ] is/are transmitted herewith.
- b. [ ] is/are not required as the application was filed with the United States Receiving Office.

9. [ ] A translation of the annexes to the international preliminary examination report

- a. [ ] is transmitted herewith.
- b. [ ] is not required as the annexes are in the English language.

10. [X] An oath or declaration of the inventor (35 U.S.C. Section 371(c)(4)) complying with 35 U.S.C. 115

- a. [ ] was previously submitted by applicant on \_\_\_\_\_  
Date
- b. [X] is submitted herewith, and such oath or declaration
  - i. [ ] is attached to the application.
  - ii. [X] identifies the application and any amendments under PCT Article 19 that were transmitted as stated in points 3(b) or 3(c) and 5(b); and states that they were reviewed by the inventor as required by 37 C.F.R. Section 1.70.
- c. [ ] will follow.

Other document(s) or information included:

11. [X] An International Search Report (PCT/ISA/210) or Declaration under PCT Article 17(2)(a):

- a. [X] is transmitted herewith.
- b. [ ] has been transmitted by the International Bureau.  
Date of mailing (from form PCT/IB/308): \_\_\_\_\_.
- c. [ ] is not required, as the application was searched by the United States International Searching Authority.
- d. [ ] will be transmitted promptly upon request.
- e. [ ] has been submitted by applicant on \_\_\_\_\_.  
Date \_\_\_\_\_

12. [X] An Information Disclosure Statement under 37 C.F.R. Sections 1.97 and 1.98:

- a. [ ] is transmitted herewith.  
Also transmitted herewith is/are:
  - [ ] Form PTO-1449 (PTO/SB/08A and 08B).
  - [ ] Copies of citations listed.
- b. [X] will be transmitted within THREE MONTHS of the date of submission of requirements under 35 U.S.C. Sections 371(c).
- c. [ ] was previously submitted by applicant on \_\_\_\_\_.  
Date \_\_\_\_\_

13. [X] An assignment document is transmitted herewith for recording.

A separate [ ] "COVER SHEET FOR ASSIGNMENT (DOCUMENT) ACCOMPANYING NEW PATENT APPLICATION" or [X] FORM PTO 1595 is also attached.

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

14. [X] Additional documents:

- a. [ ] Copy of request (PCT/RO/101)
- b. [X] International Publication No. WO 00/44483
  - i. [X] Specification, claims and drawing
  - ii. [ ] Front page only
- c. [ ] Preliminary amendment (37 C.F.R. Section 1.121)
- d. [X] Other

Petition for the Revival of an International Application for Patent  
Designating the U.S. Abandoned Unintentionally Under 37 CFR 1.137(b)

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

15. [X] The above checked items are being transmitted

- a. [X] before 30 months from any claimed priority date.
- b. [ ] after 30 months.

16. [ ] Certain requirements under 35 U.S.C. 371 were previously submitted by the applicant on \_\_\_\_\_, namely:

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

### AUTHORIZATION TO CHARGE ADDITIONAL FEES

**WARNING:** *Accurately count claims, especially multiple dependent claims, to avoid unexpected high charges if extra claims are authorized.*

**NOTE:** *"A written request may be submitted in an application that is an authorization to treat any concurrent or future reply, requiring a petition for an extension of time under this paragraph for its timely submission, as incorporating a petition for extension of time for the appropriate length of time. An authorization to charge all required fees, fees under Section 1.17, or all required extension of time fees will be treated as a constructive petition for an extension of time in any concurrent or future reply requiring a petition for an extension of time under this paragraph for its timely submission. Submission of the fee set forth in Section 1.17(a) will also be treated as a constructive petition for an extension of time in any concurrent reply requiring a petition for an extension of time under this paragraph for its timely submission." 37 C.F.R. Section 1.136(a)(3)*

**NOTE:** *"Amounts of twenty-five dollars or less will not be returned unless specifically requested within a reasonable time, nor will the payer be notified of such amounts; amounts over twenty-five dollars may be returned by check or, if requested, by credit to a deposit account." 37 C.F.R. Section 1.26(a).*

[X] The Commissioner is hereby authorized to charge the following additional fees that may be required by this paper and during the entire pendency of this application to Account No. 501-908.

[X] 37 C.F.R. Section 1.492(a)(1), (2), (3), and (4) (filing fees)

**WARNING:** *Because failure to pay the national fee within 30 months without extension (37 C.F.R. Section 1.495(b)(2)) results in abandonment of the application, it would be best to always check the above box.*

[X] 37 C.F.R. Section 1.492(b), (c) and (d) (presentation of extra claims)

**NOTE:** *Because additional fees for excess or multiple dependent claims not paid on filing or on later presentation must only be paid or these claims cancelled by amendment prior to the expiration of the time period set for response by the PTO in any notice of fee deficiency (37 C.F.R. Section 1.492(d)), it might be best not to authorize the PTO to charge additional claim fees, except possible when dealing with amendments after final action.*

[X] 37 C.F.R. Section 1.17 (application processing fees)

[X] 37 C.F.R. Section 1.17(a)(1)-(5)(extension fees pursuant to Section 1.136(a).

[ ] 37 C.F.R. Section 1.18 (issue fee at or before mailing of Notice of Allowance, pursuant to 37 C.F.R. Section 1.311(b))

**NOTE:** *Where an authorization to charge the issue fee to a deposit account has been filed before the mailing of a Notice of Allowance, the issue fee will be automatically charged to the deposit account at the time of mailing the notice of allowance. 37 C.F.R. Section 1.311(b).*

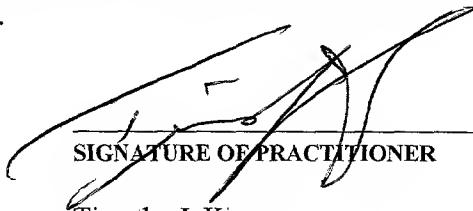
**NOTE:** *37 C.F.R. 1.28(b) requires "Notification of any change in loss of entitlement to small entity status must be filed in the application . . . prior to paying, or at the time of paying . . . issue fee." From the wording of 37 C.F.R. Section 1.28(b): (a) notification of change of status must be made even if the fee is paid as "other than a small entity" and (b) no*

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*notification is required if the change is to another small entity.*

[ ] 37 C.F.R. Section 1.492(e) and (f) (surcharge fees for filing the declaration and/or filing an English translation of an International Application later than 30 months after the priority date).

  
**SIGNATURE OF PRACTITIONER**

Timothy J. King

*(type or print name of practitioner)*

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P.O. Address

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Customer No.:



29621

PATENT TRADEMARK OFFICE

Bedford, MA 01730

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Method of Forming A Perfluorinated, Thermoplastic Hollow Fiber Module

5        The present invention relates to a method of forming a hollow fiber membrane module. More particularly, it relates to a method of making a hollow fiber module wherein the fibers and potting compound are both formed of one or more thermoplastic, perfluorinated resins.

## BACKGROUND OF THE INVENTION

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A hollow fiber membrane is a tubular filament comprising an inner diameter and an outer diameter having a wall thickness between them. Typically, this wall thickness is porous. The inner diameter defines the hollow portion of the fiber and is used to carry fluid, either the feed fluid to be filtered, or the permeating fluid, when the fluid being filtered contacts the outer surface. The inner hollow portion is typically referred to as a lumen.

Hollow fiber membrane devices have been used in many applications in the pharmaceutical, food, beverage and semiconductor industries including gas separation, reverse osmosis, ultrafiltration and particle and bacteria removal with microporous membranes. In these applications, the membrane acts as a permeable barrier, allowing the passage of the carrier fluid and some dissolved or dispensed species, and retaining other selected species due to differences in species size, permeation rates, or other physical or chemical attributes. These devices have the unique feature of requiring no support structures for the membranes as a result of their tubular geometry which makes the membrane self supporting.

In practical applications, fiber is cut or otherwise made to a specific length and a number of fibers are gathered into a bundle. A portion of one or both ends of the fiber bundle are encapsulated in a material which fills the interstitial volume between the fibers and forms a tubesheet. This process is sometimes called potting the fibers and the material used to pot the fibers is called the potting material. The tubesheet acts as a seal in conjunction with a filtration device, such as a housing or cartridge. If the encapsulation process closes and seals the fiber ends, one or both ends of the fiber bundle are cut across the diameter or otherwise opened. In some cases, the open ends of the fiber bundle are deliberately closed or sealed before potting to

prevent the potting material from entering the open ends of the fibers. If only one end is to be opened to permit fluid flow, the other end is left closed or is sealed. The filtration device supports the tubesheet and provides a volume for the fluid to be filtered and its concentrate, separate from the permeating fluid. In use, a fluid stream contacts one surface and separation occurs at the  
5 surface or within the depth of the fiber wall. If the fiber outer surface is contacted, the permeating fluid and species pass through the fiber wall and are collected in the lumen where it is directed to the open end or ends of the fiber. If the fiber inner surface is contacted, the fluid stream to be filtered is fed into the open end or ends and the permeating fluid and species pass through the fiber wall and are collected from the outer surface.

10 Most hollow fiber membrane devices are formed by potting the ends of the bundle of fibers in an adhesive such as an epoxy or urethane resins. These membranes have been found to be deficient in purity as well as in their capability for resisting aggressive or corrosive chemicals and solvents. For example, the organic solvent-based solutions used in wafer coating processes in the microelectronics industry will dissolve or swelling and weaken epoxy or urethane potting  
15 materials. Additionally, the high temperature stripping baths in this industry consist of highly acidic and oxidative materials which destroy such potting compounds.

More recently, it has been proposed to pot these membranes in a thermoplastic resin, in particular to use thermoplastics such as polyethylene or polypropylene and in some instances ultrahigh molecular weight polyethylene. See U.S. 5, 695, 702. While this is more acceptable than the previous epoxy or urethane method, it is limited in the membranes and resins which one may select for use in its method. Unlike epoxy or urethane that have relatively low viscosity and therefore flow easily around and between the fibers to be potted, thermoplastic materials are of higher molecular weight and higher viscosity and are difficult to cause to flow around and between such fibers. Moreover, even when one is able to achieve some flow, the flow is not uniform,  
20 leading to irregularities, voids and gaps in the potting. Additionally, these molten thermoplastic materials often have detrimental effects on the fibers themselves, causing fibers to collapse, shrink or melt. This has led to practitioners to adopt various complicated schemes to overcome these problems.

There exists a class of thermoplastic materials known as perfluorinated thermoplastic  
30 polymers. These materials are known by various chemical terms such as poly(TFE-co-PFAVE) resins ("PFAVE"). These materials are also commonly referred to as PFA, MFA or FEP resins.

These materials have the chemical inertness similar to that of PTFE resin, but unlike PTFE resin, are thermoplastic and therefore capable of being molded and formed into various shapes easily and economically. Hollow fibers made from such perfluorinated resins have been made. Such fibers and methods for making them are disclosed in U.S. 4, 990,294 and 4,902,456.

5 None of the methods to date have demonstrated a capability of potting perfluorinated hollow fibers with a perfluorinated polymer potting resin. As is typical with such polymers, they inherently are difficult to have bond to themselves or other materials.

A hollow fiber membrane device formed completely of perfluorinated thermoplastic resins, including the fibers, potting resin and module components such as the end caps and cartridge 10 housing would be highly desirable in that it would provide a completely inert system, especially at elevated temperatures which would be capable of resisting aggressive or corrosive chemicals and organic solvents, which would provide the highest level of purity available as such materials are inherently clean and tend not to have a high level of extractables and which because it is formed of thermoplastic materials could be formed relatively easily and efficiently. The present invention 15 provides such a method and a product made by that method.

#### IN THE DRAWINGS

Figure 1 shows a perspective diagram of an apparatus useful in making an array of hollow 20 fiber membranes in accordance with one preferred embodiment of the present invention;

Figure 2 is a top view of a finished array of hollow fiber membranes made in accordance with the process of Figure 1;

Figure 3 is a schematic view of an apparatus used in the making of the hollow fiber membrane tubesheet according to a preferred embodiment of the present invention;

25 Figure 4 shows a detailed view, in perspective, showing the spiral winding sealing of a tubesheet according to a preferred embodiment of the present invention;

Figure 5 shows a perspective view of a device for accomplishing the post-formation heat 30 treating step according to a preferred embodiment of the present invention;

Figure 6 shows a sectional view of a hollow fiber membrane module in a cartridge housing constructed in accordance with a preferred embodiment of the present invention.

### SUMMARY OF THE INVENTION

5 It has been found that by using a perfluorinated resin, in particular a thermoplastic perfluorinated potting resin which has a lower peak melting temperature than the membrane and a reasonably low melt viscosity, integral potting of the hollow fiber membranes can be achieved. The combination of these two characteristics of the potting resin allows for longer contact time between the molten resin and the fibers to attain more complete encapsulation and sealing of the fibers

10 without damage to the fibers or collapse of their lumens. An assembled bundle of fibers and potting resin are heated to a temperature above the peak melting point of the potting resin and below the peak melting point of the fibers, in order to cause the potting resin to flow and fill any gaps or voids between the fibers which may be present. Long processing times can be used, if necessary, during this heating step to ensure adequate sealing without risk of fiber damage or lumen collapse. The resultant potted fiber bundle may then be assembled into a cartridge housing made of one or more thermoplastic perfluorinated resins, and sealed with one or more end caps also formed of one or more thermoplastic perfluorinated resins to form an integral all thermoplastic perfluorinated resin based module. Alternatively, other inert fluorinated resins may be used as the housing or end caps, such as PTFE resin, in order to create an all fluoropolymer module.

15

20 It is an object of the present invention to provide a method of forming a thermoplastic, perfluorinated resin hollow fiber bundle comprising the steps of contacting a plurality of hollow fibers made of a thermoplastic, perfluorinated resin with a molten thermoplastic perfluorinated potting resin wherein the potting resin has a peak melting point below that of the hollow fibers and a relatively low melt viscosity so as to allow the potting resin to flow around the hollow fibers to

25 form a bundle, cooling the bundle, heating the bundle again to a temperature above the peak melting point of the potting resin but below the peak melting point of the hollow fibers for a period of time sufficient to create a fluid-tight seal between the fibers and the potting resin.

It is an object of the present invention to provide a method of forming an all thermoplastic, perfluorinated resin hollow fiber module comprising steps of contacting a plurality of hollow fibers made of one or more thermoplastic perfluorinated resins with a molten potting resin made of one or more thermoplastic perfluorinated resins in order to form a bundle, the potting resin having a peak

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melting point below that of the hollow fibers and which is applied to the hollow fibers at a temperature below the peak melting point of the hollow fibers, cooling the bundle, heating the bundle to a temperature at or above the softening point of the potting resin but below the peak melting point of the hollow fibers for a period of time sufficient to create a fluid-tight seal between  
5 the fibers and the potting resin.

It is an object of the present invention to provide an all thermoplastic perfluorinated resin hollow fiber module formed by forming a bundle of hollow fibers made of one or more thermoplastic perfluorinated resins and potting at least one of the two ends of the fibers with a potting compound formed of one or more thermoplastic perfluorinated resins, cooling the bundle, heating the bundle  
10 to a temperature at or above the peak melting point of the potting resin but below the peak melting temperature of the fibers for a period of time sufficient to cause the potting resin to flow around and between said fibers in order to create a fluid-tight seal between the fibers and the potting resin removing a portion of the potting compound in order to open the interior of the fibers to the exterior environment, inserting the bundle into a housing having a first and second end and sealing said  
15 bundle into said housing, applying an end cap to the first and second ends of the housing in order to form an integral, all perfluorinated thermoplastic hollow fiber module.

These and other objects of the present invention will be made clear from the following description and examples.

20

## DETAILED DESCRIPTION OF THE INVENTION

A method of making an all perfluorinated, thermoplastic hollow fiber module in accordance with the present convention can be accomplished through the use of one or more thermoplastic perfluorinated resins, both as the hollow fiber membrane and potting compound as well as the  
25 components of the module itself.

One aspect of the present invention is to eliminate voids and gaps in the potting material via the use of a post-potting heating treatment. This treatment allows for the potting resin to be melted such that it flows between the fibers and eliminates any gaps or voids that may exist and to allow for adequate bonding of the fibers to the potting materials.

30

The method of fabricating a hollow fiber membrane device according to a preferred embodiment of the present invention begins with the formation of the thermoplastic perfluorinated

resin based hollow fiber membranes such as are taught in U.S. 4, 990, 294 and U.S. 4, 902, 456, and co-pending application US Patent Application 60/117,852 and 60/117,854, filed January 29, 1999, the teachings of which are incorporated herein in their entireties.

The fibers are then arranged and then formed into a bundle by applying one or more streams of molten potting compound, which consists of a perfluorinated thermoplastic resin or blend of such resins, to one or more ends of the individual fibers, while such fibers after having the stream or streams applied are wound upon themselves or a mandrel to form the bundle.

The potting compound, the fibers and to extent it may be used, the mandrel, may be made of one or more thermoplastic perfluorinated resins such as poly(TFE-co-PFAVE) resins. These resins include poly(tetrafluoroethylene-co-perfluoro(alkylvinylether)) resins, often called by the term PFA or MFA or poly(tetrafluoroethylene-co-hexafluoropropylene) resin, often called FEP or blends thereof. When using a poly(tetrafluoroethylene-co-perfluoro(alkylvinylether)) resin, the alkyl group is preferably either a methyl or propyl group or blends thereof.

Such resins are commercially available. For example, one may use TEFLON® PFA resin available from E.I. duPont de Nemours of Wilmington, Delaware or HYFLON® MFA resin available from Ausimont USA of Thorofare, New Jersey or NEOFLON® PFA available from Daikin Industries of Japan.

The perfluorinated thermoplastic resin selected for the fibers should have a peak melting point that is greater than the peak melting point of the perfluorinated thermoplastic resin selected for the potting compound. This allows for adequate bonding of the fibers to the potting material without detrimental effects to the fiber caused by the temperature of the potting material.

Fibers may be made from a poly(tetrafluoroethylene-co-perfluoro(alkylvinylether)) wherein the alkyl group is mainly methyl, such as HYFLON®620 resin available from Ausimont USA of Thorofare, New Jersey which has a peak melting point of about 285°C or from a poly(tetrafluoroethylene-co-hexafluoropropylene) resin, such as TEFLON®FEP 100 resin available from E.I. duPont de Nemours of Wilmington, Delaware which has a peak melting point of about 270°C.

Typically, the potting compound has a peak melting point at least 5 °C below that of the fibers. Preferably, the peak melting point of the potting compound is at least about 10°C below that of the fibers. More preferably, it has a peak melting point of at least about 25°C below that of

the fibers. For example, for fibers formed of a perfluorinated thermoplastic resin such as HYFLON® 620 resin, they have a peak melting point of about 285° C. The potting compound will be a perfluorinated thermoplastic resin or blend of such resins having a peak melting point of less than about 275°C, preferably having a peak melting point of from about 230°C to about 275°C.

5 More preferably, the potting resin has a peak melting point of less than about 260°C.

Likewise, the potting compound should have a relatively low melt viscosity.

Typically, the melt flow index(MFI) (MFI as defined by A. S. T. M. D2116, at 372°C and 5kg.load) of the perfluorinated thermoplastic resin or resins used in the potting compound should be greater than 100g/10 min. Preferably the melt flow index is greater than 150 g/10 min. More preferably, the melt flow index is greater than 200g/10 min. It has been found that the higher the melt flow index, the better the resultant product. A resin or resins may be used which have a lower melt flow index than the values described above, however one will find that the process becomes more difficult and the seal between the fibers and the potting compound may become incomplete.

One example of a poly(tetrafluoroethylene-co-perfluoro(alkylvinylether) useful as a potting material is sold as HYFLON®940 AX resin from Ausimont USA Inc of Thorofare, New Jersey. It has a peak melting point of from about 250°C to 260°C and a melt flow index of about 130g/10 mins.. An example of a suitable low viscosity poly(tetrafluoroethylene-co-hexafluoropropylene) resin useful as a potting compound is taught in U.S. Patent 5, 266, 639, especially those at the lower end of melt temperatures.

20 The following is a description of a preferred method of making and using the present invention.

A flat substantially parallel array of these fibers is prepared. One may do so by forming a weave of fibers with the fibers either being the weft or warp and the other component being one or more fibers or threads or by forming a tape of fibers, a mat of fibers, or by any other known means 25 for forming such an array.

Preferably, the array is formed according to the teachings of U.S. 5, 695, 702, the teachings of which are incorporated by reference herein. According to that reference, the fibers are first formed into an array by arranging individual fibers substantially parallel to each other. The fibers may be spaced apart from each other or if desired, they may be touching each other. Which arrangement is selected depends upon the packing density desired in the final module. Typically,

one wishes to have a packing density of between 40 and 70%, more preferably between 45 and 65%. Packing density is defined as the ratio, expressed as a percentage, of the total cross-sectional area of all the fibers in a bundle to the cross-sectional area of the final potted fiber bundle. If the packing density is too low, then filtration efficiency is compromised. If the density is  
5 too high, then there is the danger that incomplete potting will occur or that filtration efficiency will be compromised as the fluid will have difficulty reaching the inner fibers of the bundle.

In order to reduce contamination of the fibers or the finished module, any means used to make the array should be confined to areas that will normally be trimmed from the finished hollow fiber module.

10 The array may be fabricated by either winding the length of the array upon itself or upon a mandrel. The mandrel, if chosen, is preferably circular in cross section, however other cross sections, such as oval, square, rectangular or polygonal may also be used. The circumference of the mandrel is chosen to be an integer multiple of the desired length of the hollow fiber member elements which form the finished array. When using the mandrel, it is preferably driven by a  
15 mechanical device such as a controller capable of controlling both rotational speed and tension applied to the array. The wound hollow fiber membrane array is arranged such that it is fed as a single layer of fibers, with the windings being substantially parallel to each other either in contiguous contact with or spaced uniformly apart from one another.

The array is fabricated by winding a continuous length of hollow fiber membrane 11 on a  
20 rotating mandrel 12 having a circular cross section as shown in Figure 1. The circumference of the mandrel is chosen to be an integer multiple of the desired length of the hollow fiber membrane elements which are to comprise the finished array. The mandrel is driven by a controller 13 capable of controlling both the rotational speed of the mandrel and the tension applied to the hollow fiber membrane. The controller includes a fiber feed mechanism 14 which moves a pulley  
25 15 parallel to the central axis of the mandrel and guides the hollow fiber membrane as it is being wound to control the spacing between adjacent fiber segments. The wound hollow fiber membrane is arranged in a single layer, with the windings being substantially parallel to one another either in contiguous contact with, or spaced uniformly apart from, one another.

When the appropriate length of hollow fiber membrane 11 is accumulated on the mandrel  
30 12, the controller 13 stops the winding operation and one or more strips of an adhesive tape 22 (Figure 2) are applied to the outer surface of the hollow fiber membrane segments positioned along

the length of the mandrel in an orientation parallel to its axis of rotation and perpendicular to the central axes of the individual hollow fiber segments. More than one strip of tape can be used, the circumferential spacing between strips being equal to the desired axial length of the membrane fibers in the array 21. The tape extends from the first hollow fiber membrane segment wound on  
5 the mandrel to the least and preferably extends about 1 cm. beyond each end of the fiber array.

A cutting guide (not shown) may be used to slit the hollow fiber membrane segments along the middle of the entire length of the tape 22 such that the hollow fiber membranes 11 remain joined together by the now halved strip of tape. In this manner, one or more hollow fiber membrane arrays 21 are produced, with the fiber elements being secured to one another at their  
10 ends by the tape thereby making it easily removed from the mandrel 12. It should be noted that, in this discussion, the edges 23 of the rectangular hollow fiber membrane array 21 are defined as the two surfaces formed by the end portions of the individual hollow fiber membrane elements comprising the array; the ends 24 of the array are defined by the outermost surfaces of the first and last hollow fiber membrane elements in the array. Figure 2 shows in the plan view an array formed  
15 in accordance with the above procedures. In cases where one fiber array does not contain a sufficient number of hollow fibers to fabricate a hollow fiber membrane module of the desired membrane area, the arrays may be spliced together end-to-end by means of an adhesive or other bonding mechanism to form a larger array. Any number of arrays may be so spliced together in the manner described above to form a larger array having tape extensions at the edges of both  
20 ends of the array.

The next operation in the fabrication of a hollow fiber membrane module is the winding of the fiber array into a bundle and the corresponding formation of a pair of tubesheets 43 at one or more of the edges 23 of the array 21. This process is shown schematically in Figures. 3 and 4. An extruder, preferably a single screw extruder, 31 is used to feed a thermoplastic sealing polymer to  
25 a dual slot extrusion die 32 which produces two polymer extrusions 35 in the form of a stream. A suitable length of thermoplastic tube 41 is mounted on a removable winding mandrel 42 positioned below the extrusion die, with the rotational axis of the mandrel being parallel to a line connecting the two outlets of the extrusion die. Stepper motors (not shown) are used to adjust the speed of rotation and distance between the mandrel and the die. A set of gas heaters 33 mounted on a  
30 retractable slide (not shown) is used to preheat the tube 41 prior to the fabrication of the

tubesheets. The functions of the various elements described above are regulated by a programmable, microprocessor-based controller 34.

In order to maintain the molten thermoplastic polymer extrusion 35 from the die at a uniform temperature, it is preferred to operate the extruder 31 at a constant speed. Maintaining a uniform fiber spacing and tubesheet width requires that the fiber feed rate remain constant and that the distance between the extrusion die and contact point of the polymer extrusion and tubesheet 43 remain constant. The previously described controller 34 in conjunction with the apparatus discussed above accomplish this result with feed-back control mechanisms known to those skilled in the art.

Before the winding of the array 21 and the formation of the tubesheets 43, the tube 41 must be pre-heated using the heaters 33. This step is necessary to obtain a good bond between the tubesheet and the tube. Rotation of the winding mandrel 42 and the tube is begun and the gas heaters are activated such that a hot gas stream impinges on the portions of the tube where the tubesheets will be formed. After a suitable time, the heaters are removed and the polymer extrusions 35 are applied to the tube.

Following the accumulation of approximately a one-half turn of the polymer extrusions 35 on the tube 41, the leading edge of the hollow fiber membrane array 21 is positioned under and parallel to the tube with the adhesive side of the extended strip of tape 22 facing the tube. The tape is then brought into contact with the tube outboard of the tubesheets 43 and allowed to wind up on the tube as the rotational speed and position of the winding mandrel 42 and tube are adjusted by the process controller 34. A slight tension is maintained on the hollow fiber array to keep the fibers in contact with the polymer extrusions. As the trailing edge of the array is wound up, the tape extensions are fastened to the previous fiber layer to form a fiber bundle 44. Application of the polymer extrusions may be terminated after the entire array is wound about the mandrel. Alternatively, the tubesheets may be built up to a larger diameter depending on the requirements of the rest of the module assembly process. In this case, the rotation of the winding mandrel continues as the tubesheets are allowed to cool.

After formation and cooling of the hollow fiber bundle, a post-formation heating step is performed. In this step, the potted ends are heated to a temperature below the peak melting point of the hollow fiber membranes. The temperature selected is above the peak melting point of the potting resin, but below the peak melting point of the hollow fiber membranes. For example, the

heating temperature may range from 250°C to 300°C. An alternative means for describing this temperature is that it should be at least 10°C below the peak melting point of the fibers, preferably at least 15°C and more preferably, at least 25°C below the peak melting point of the fibers. This step causes the potting material to melt, and flow between the fibers in order to eliminate any voids  
5 or gaps which may exist. One advantage to using the post-formation heating step is that the criticality of the initial extrusion step is not as important as the post-formation heating step allows for the elimination of any voids formed during the extrusion step. This is of particular interest and importance in that the extrusion step often is very slow and laborious.

The time required for the post-formation heating step will vary widely depending upon  
10 such factors as the peak melting point of the potting compound and the fibers, the viscosity/melt flow index of the potting compound, the perceived level of voids or gaps which must be eliminated, etc.. Typically, the process requires from about 1 hour to about 24 hours. It has been found that the longer one is able to perform the heating step, without damage to the fibers, the better the result. Preferably, the heating step occurs from about 3 to about 12 hours, more preferably from  
15 about 5 to about 8 hours.

Preferably, the heating step occurs with the tubesheet in a vertical orientation with one end being treated at a time. Further, the end or ends may be treated in a horizontal orientation provided that little or no slumping occurs. This is particularly possible with higher viscosity or lower melt flow index potting compounds. Preferably, the heat treatment occurs prior to the formation of the  
20 filtration cartridge. Alternatively, the heat treatment may occur after the tubesheet has been inserted into the housing or end caps. In this embodiment, the heat treatment acts both as a mechanism for removing any gaps or voids and as the sealing mechanism for attaching the bundle to the housing or end caps. Additionally, while it is preferred that the heating step occur before the lumens of the fibers are opened, one can perform the step after the lumens have been opened  
25 provided that some means is used to maintain the opened ends of the lumens.

Figure 5 shows a preferred embodiment of the post-formation heat treatment. As shown, one end 43A of the tubesheet 43 is placed into a cup-like holder 50 which has a depth and diameter about the same as the potted end 43A. The holder 50 is mounted into a recess 51 formed in a metal heating block 52. The block 52 is heated with one or more electrical heating  
30 bands 53. The holder 50 is preferably made of a thermally conductive material, such as metal or ceramics may also be used.

Alternatively, one may use other means of providing the heat treatment. For example, one may use an oven including but limited to radiant, convection or microwave ovens in lieu of the holder/block arrangement above. The use of a holder or holders in an oven or other heating means may be desirable. The holders contain the area into which the potting material may flow. This 5 concentrates the flow to the areas between the fibers. Additionally, one may use two holders at the same time in order to treat both ends of the tubesheet simultaneously whether the heating is via a heating block arrangement, an oven or other well known means in the art.

Either one or both of the end portions of the sealed fiber bundle can be trimmed to expose the fiber lumens and further machining may be performed to provide a means for sealing the fiber 10 bundle into a suitable housing or the fiber bundle may be contoured to provide details suitable for thermoplastically bonding it to the components of a pressure housing of the same or similar resin material in order to produce a hollow fiber module.

Figure 6 shows the details of the fiber bundle 44 and corresponding tubesheets 43 (labeled in this figure as 43b and 43t to represent the bottom and top orientations shown in the drawing) after the postheating step which have been assembled into a hollow fiber module. 15 Preferably, this module is formed of the same resin as either the hollow fibers or the potting material in order to create an all perfluorinated resin device. Preferably, this can be accomplished by employing conventional methods of fusion bonding of plastic components. Alternatively, the housing may be made of a similar and compatible resin such as PTFE. However, when using such a resin, especially PTFE where the resin is not thermoplastic, other means of sealing the tubesheet 20 to be housing, such as O-rings or mechanical seals, must be used. After fabrication of the bundle 44 is completed, the bottom tubesheet 43b is bonded to an inside cap 71. During use, the filtrate collected at that tubesheet is directed to a top end cap 72 through the tube 41. The housing shell and the top tubesheet 43t of the bundle are simultaneously bonded to the top end-cap. Finally, an 25 outside end-cap 73 is bonded to the bottom of a housing shell 74. Suitable connectors are also added to provide means of connecting the module to a feed and to an effluent line. By these means an integral, module free of O-ring seals can be produced.

Although the embodiments heretofore described have involved the formation of an array of 30 hollow fiber membrane elements prior to sealing with a molten thermoplastic, the principles of the present invention are equally applicable to the formation of an array one fiber at a time. In this instance, a mechanical "pick-and-place" mechanism would be employed to a single fiber to a

location beneath an extruder such that the polymer extrusion simultaneously creates a seal and forms the array by repeating this process until an array of desired size is created.

Various other configurations of pre-constructed fiber arrays are also possible. For example, the fibers do not have to be positioned perpendicularly to the longitudinally axis of the array. Fiber array bundle arrangements may also vary in that single-ended tubesheets for short length membrane modules may be fabricated as well as multiple tubesheets. In the former case, the hollow fiber elements must be sealed at the opposite end of the tubesheet. In the latter case, intermediate tubesheets, used primarily as support members, may or may not form integral seals around the hollow fiber membranes for particularly long modules. Furthermore, multiple bundles may be produced simultaneously using integrally sealed, multiple tubesheets which are subsequently cut to form individual fiber bundles. Still further, planar laminated arrays are also contemplated to be within the purview of this invention, in which case rectangular fiber arrays may be mounted on top of one another. The use of woven arrays is also possible.

An alternative method of making a product according to this invention is to form a mat or flat array of fibers using the thermoplastic, perfluorinated resin as the tape binder for the array and then rolling the array into a cylindrical or other desired shape and subjecting the ends to the post-formation heating step to form an integral, sealed tubesheet. For example, a mat of fibers in the form of an array would have a stream of potting material extruded along two of its portions of its length, preferably in parallel and spaced apart from each other at a desired distance equal to that of the finished tubesheet. Alternatively, a mat can be formed using the potting material in the form of a solid tape which is woven between the adjoining fibers. Further, one can adhere or secure individual fibers to two parallel strips of thermoplastic, perfluorinated resin.

With regard to the manufacture of a finished module, other means for sealing the bundle to the module may be employed, as for example, with the use of O-rings (preferably formed of a perfluorinated resin such as thermoplastic perfluorinated resin or PTFE resin,).

Additional modifications will become apparent to those of skill in the art without departing from the scope of the present invention as defined in the accompanying claims.

### Example 1

Microporous thermoplastic perfluorinated hollow fiber membranes made from Ausimont's MFA 620 resin according to the teachings of US Patents 4, 990, 294 and 4, 902, 456 were used for potting in this example. The peak melting temperature of the fibers as measured by differential scanning calorimetry (DSC) was 289°C. The outside diameter of each fiber was 1000 microns and the inside diameter was 600 microns. Porosity was approximately 65%. The potting resin used was a thermoplastic perfluorinated resin available from Ausimont as MFA19405/13 resin. The peak melting temperature of this resin was 258°C and its melt flow rate (MFI at 5kg, 372°C as described by ASTM D 2116) was 124g/10 minutes.

Approximately 90 strands of the above fibers, each being about 15 cm in length, were arranged in a parallel array and taped together near both ends of the fiber to form a fiber mat. A method similar to that described in U.S. 5, 695, 702 was used to extrude two molten streams of the potting resin described above in a perpendicular direction onto the mat. The streams were spaced about 9 cm apart and were each about 2.5 cm in width having a thickness of about 0.075 cm. The stream die temperature was set at 335°C. The mat/molten resin streams combination was spirally wound on a poly(tetrafluoroethylene-co-hexafluoropropylene) tube into a cylindrical shaped bundle with a pair of potted ends. It was observed that the molten potting resin also bonded to the FEP tube.

After the potted ends were cooled, the bundle was removed and inspected. One could visually observe a number of voids and bubbles in the potting surrounding the fibers. Adhesion strength was excellent. The fibers could not be pulled out of the potting compound.

Following inspection, excess fibers and tubing beyond the potted ends were trimmed off to be readied for post-extrusion heat treatment. One of the ends was then placed in a cylindrical cup-shaped metal holder with depth and diameter approximately the same dimensions as the potted end. The holder with the potted end was then fitted into a cut-out in a metal heating block. The block was heated with electrical heating bands and its temperature was controlled at 280°C. The sample was heated for about one hour at that temperature. This procedure was repeated for the opposite end of the bundle. After completion of the post-extrusion heat treatment, the ends were machined to until the lumens of the fibers were exposed. The fibers were observed to be bonded together on their shell side by the potting resin and no visible voids were observed. Adhesion strength was the same as before the heating treatment.

The potted sample was tested for potting integrity by placing and sealing both ends via O-rings inside a clear plastic housing. Iso-propyl-alcohol (IPA) was used to wet the fibers and fill the inside of the housing, making sure that all of the potted areas were covered with IPA. Both ends of the housing were capped with one of the caps having an inlet for pressurized air. Air pressure was 5 slowly introduced and increased via the inlet on the one end. Simultaneously, the inside of the holder was examined visually for air bubbles originated from the potting area. No bubbles were observed at either end up to at least 15 psi, indicating integral potting had been achieved.

10 What we claim:

A copy of the original application filed in the United States Patent and Trademark Office is available for inspection at the Patent and Trademark Information Center, 1100 Connecticut Avenue, N.W., Washington, D.C. 20591.

CLAIMS

1. A method of forming an all thermoplastic, perfluorinated resin hollow fiber module comprising the steps of:

5                   contacting a plurality of hollow fiber membranes made from one or more thermoplastic, perfluorinated resins with one or more molten thermoplastic, perfluorinated potting resins to form a substantially parallel array of said membranes;

10                  said one or more potting resins being heated sufficiently above their peak melting point but at or below the peak melting point of the membranes such that they are applied to said membranes at a contact temperature which causes said one or more potting resins streams to flow around said hollow fiber membranes to form a bundle of hollow fiber membranes;

15                  cooling said bundle;

15                  heating said bundle to a temperature below the peak melting point of the hollow fibers and above the peak melting point of the one or more potting resins for a period sufficient to form a fluid-tight seal between the one or more potting resins and the hollow fiber membranes.

20                  2. The method of claim 1 wherein the peak melting point of the one or more potting resins is at least 5°C below that of the hollow fiber membranes.

25                  3. The method of claim 1 wherein the peak melting point of the one or more potting resins is at least 10° C below the peak melting point of the hollow fiber membranes.

25                  4. The method of claim 1 wherein the one or more thermoplastic, perfluorinated resins of the hollow fiber membranes and the potting resins are selected from the group consisting of homopolymers, copolymers, blends of one or more homopolymers, blends of one or more copolymers and blends of one or more homopolymers and copolymers of perfluorinated resins.

5. The method of claim 1 wherein the one or more thermoplastic, perfluorinated resins of the hollow fiber membranes and the potting resin are selected from the group consisting of poly(TFE-co-PFAVE) resins and blends thereof.

5 6. The method of claim 1 wherein the bundle is heated to a temperature at or above the peak melting point of the one or more potting resins.

7. The method of claim 1 wherein the plurality of hollow fiber membranes is formed prior to contacting said membranes with said potting resins by forming said membranes together in a  
10 contiguous relation.

8. The method of claim 1 wherein the array is formed prior to contacting said membranes with said potting resins by forming said membranes together in a spaced apart relation.

15 9. The method of claim 1 wherein the potting resin is a thin stream deposited in a defined zone near one end of said membrane array.

10. The method of claim 1 further comprising the step of contacting a second thin stream of potting resin near an opposite end of said array of membranes.  
20

11. The method of claim 1 further comprising the steps of forming a substantially parallel array of said membranes and subsequently spirally winding said array about an axis which is substantially parallel to a longitudinal axis of said membrane array while simultaneously applying said potting resin to the array of membranes to form circular bundle of fibers having at least one  
25 potted end.

12. The method of claim 11 further comprising the step of continuing to apply said potting resin after said circular bundle is formed to create a tubesheet of predetermined diameter about at least one end of said hollow fiber membranes.  
30

13. The method of claim 1 further comprising the step of cutting the at least one potted end of the bundle orthogonally to the longitudinal axis of said hollow fiber membranes to form said bundle with at least one flat end surface having exposed lumens.

5 14. The method of claim 13 further comprising the step of mounting said bundle into a cartridge housing.

15. The method of claim 14 wherein the bundle is mounted in said housing by fusion bonding.

10 16. A. method of making a hollow fiber membrane cartridge comprising:

a. forming a plurality of hollow fiber membranes formed of one or more thermoplastic perfluorinated resins into a substantially parallel arrangement wherein the fibers are arranged in parallel arrangement along a length of the fibers; then

b. winding the plurality of hollow fibers about an axis which is substantially parallel to the length of the hollow fiber membranes so as to form a bundle having two bundle ends;

c. simultaneously with step (b), extruding a molten stream of a perfluorinated thermoplastic resin having a peak melting point at least 5°C below the peak melting point of the hollow fiber membranes and having a melt flow index of 100 g/10mins. or greater and directing said resin onto at least one of the two bundle ends to thereby pot one or more ends in said resin;

d. cooling the bundle;

e. heating the bundle at the one or more potted ends to a temperature at or above the peak melting point of the resin of the stream but below the peak melting point of the hollow fibers; and

f. exposing the lumen ends of the hollow fiber membranes at one or more potted bundle ends to communicate with the exterior of the bundle.

25 17. The method of claim 16 wherein both ends of the bundle are potted with the molten stream 30 of the perfluorinated thermoplastic resin.

18. The method of claim 16 wherein both ends of the bundle are potted with the molten stream of the perfluorinated thermoplastic resin and wherein both ends of the bundle are exposed so that the lumen ends of the hollow fiber membranes can communicate with the exterior of the bundle.

5 19. A hollow fiber membrane cartridge including a bundle of potted hollow fiber membranes, made by the method of claim 1.

20. A hollow fiber membrane cartridge including a bundle of potted hollow fiber membranes, made by the method of claim 16.

10

21. The method of claim 16 further comprising the steps of:

g. inserting the bundle into a housing for the bundle having a first and second end and a cylindrical housing interior being suitably shaped to contain the membrane bundle, a first means for sealing the first end of the bundle to the interior of the housing adjacent its first end, a second means for sealing the second end of the bundle to the interior of the housing adjacent its second end, the housing having one or more means for dividing the bundle into at least two regions including a shell side space exterior to the portion of the bundle between the potted ends and a space including the lumens; then

h. applying a first end cap adjacent the first end of the housing to seal the first housing end; then

i. applying a second end cap adjacent the second housing end so as to seal the second housing end; and

j. providing a shell side access in the housing and at least one access in at least one of the first or second end caps.

25

22. A hollow fiber membrane cartridge including a bundle of potted hollow fiber membranes made by the method of claim 21.

23. A method according to claim 16 wherein the potting compound has melt flow index of from about 100 to about 200 g/10mins..

30

24. A method of forming a hollow fiber module comprising the steps of:

forming a substantially parallel array of hollow fiber membranes, wherein said membranes are formed of one or more thermoplastic perfluorinated resins,

5 forming one or more strips of potting material formed one or more thermoplastic perfluorinated resins along one or more portions of the array, wherein the potting material has a peak melting point at least 5°C below that of the hollow fiber membranes

winding the array upon itself in order to form a bundle,

heating said bundle to a temperature below the peak melting point of the hollow fibers and above the peak melting point of the one or more strips of potting material for a period sufficient to form a fluid-type seal between the potting material and the hollow fiber  
10 membranes.

25. The method of claim 24 wherein the one or more thermoplastic perfluorinated resins of the hollow fiber membranes and the potting material are selected from the group consisting of poly(tetrafluoroethylene-co-perfluoro(alkylvinylether)), poly(tetrafluoroethylene-co-  
15 hexafluoropropylene) and blends thereof.

26. The method of claim 24 wherein the one or more strips of potting material is applied to the array of fibers as a molten stream.

27. The method of claim 24 wherein the one or more strips of potting material is applied to the array of fibers as a solid, preformed tape.

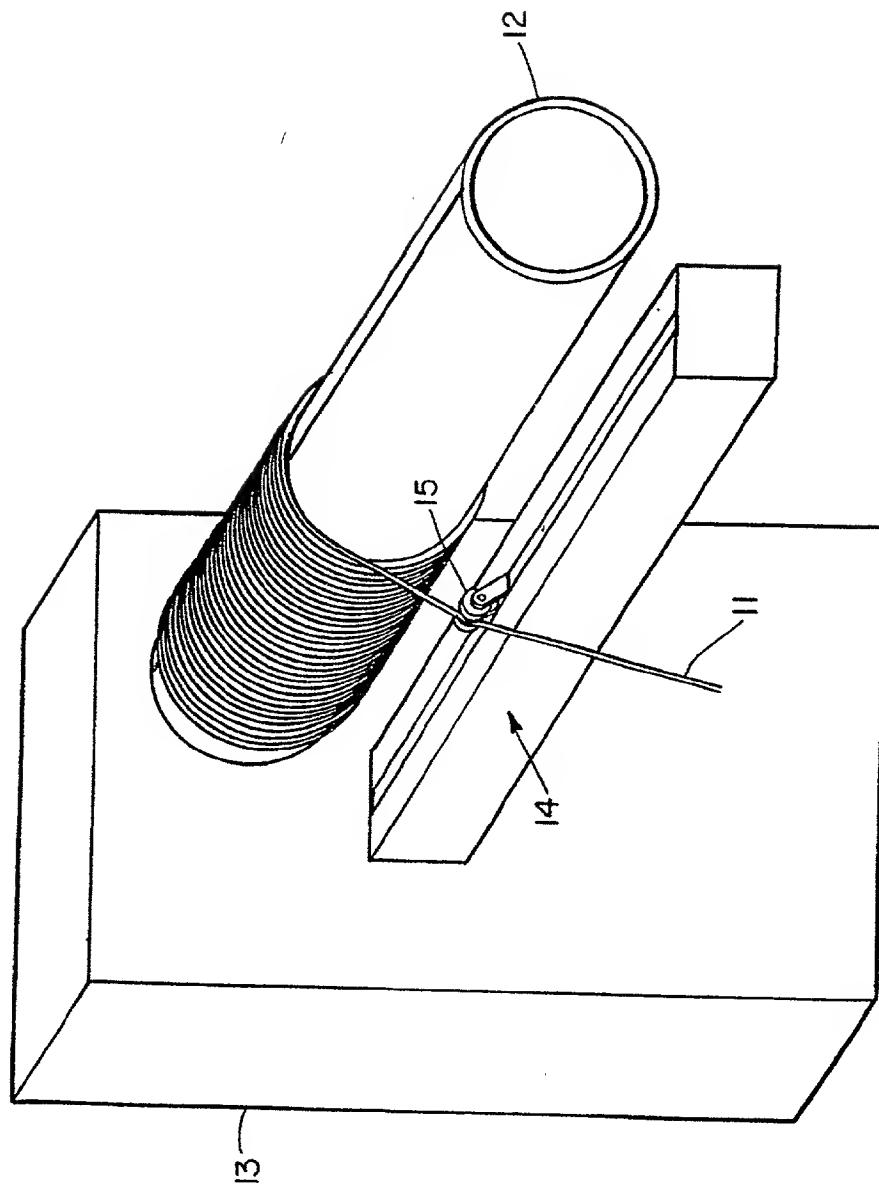


FIG. 1

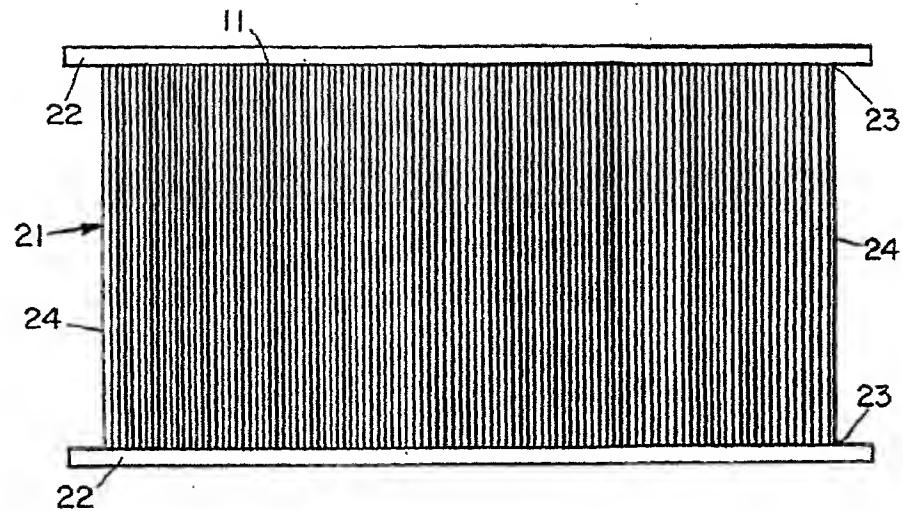


FIG. 2

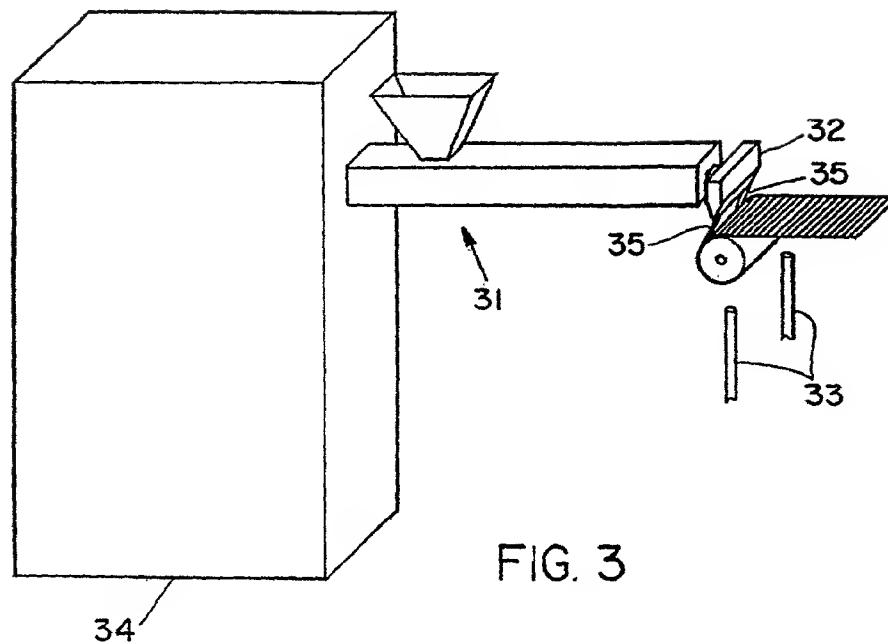


FIG. 3

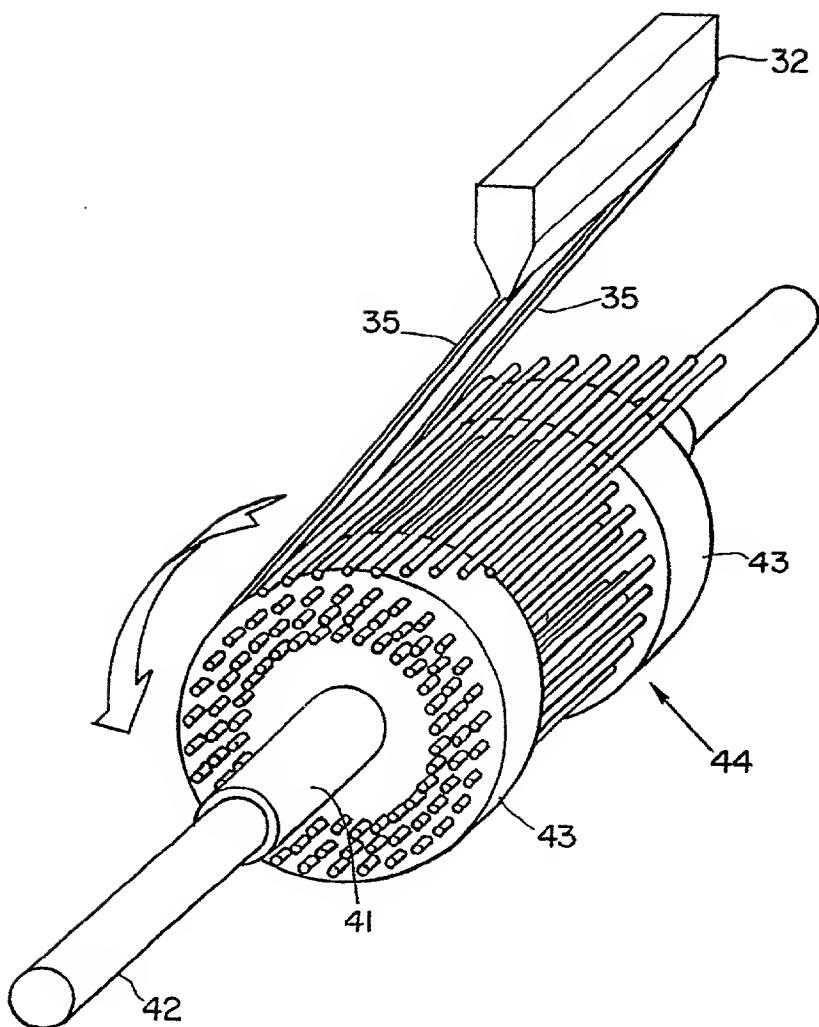


FIG. 4

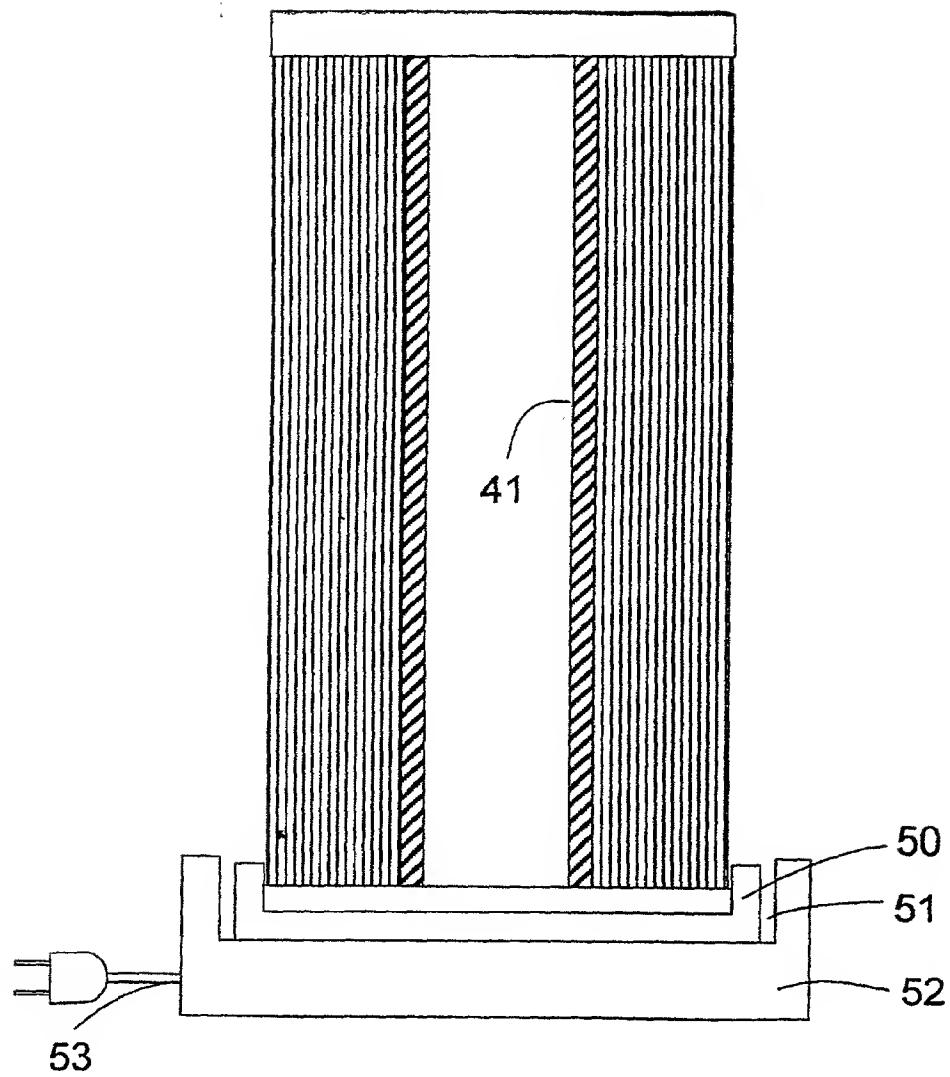
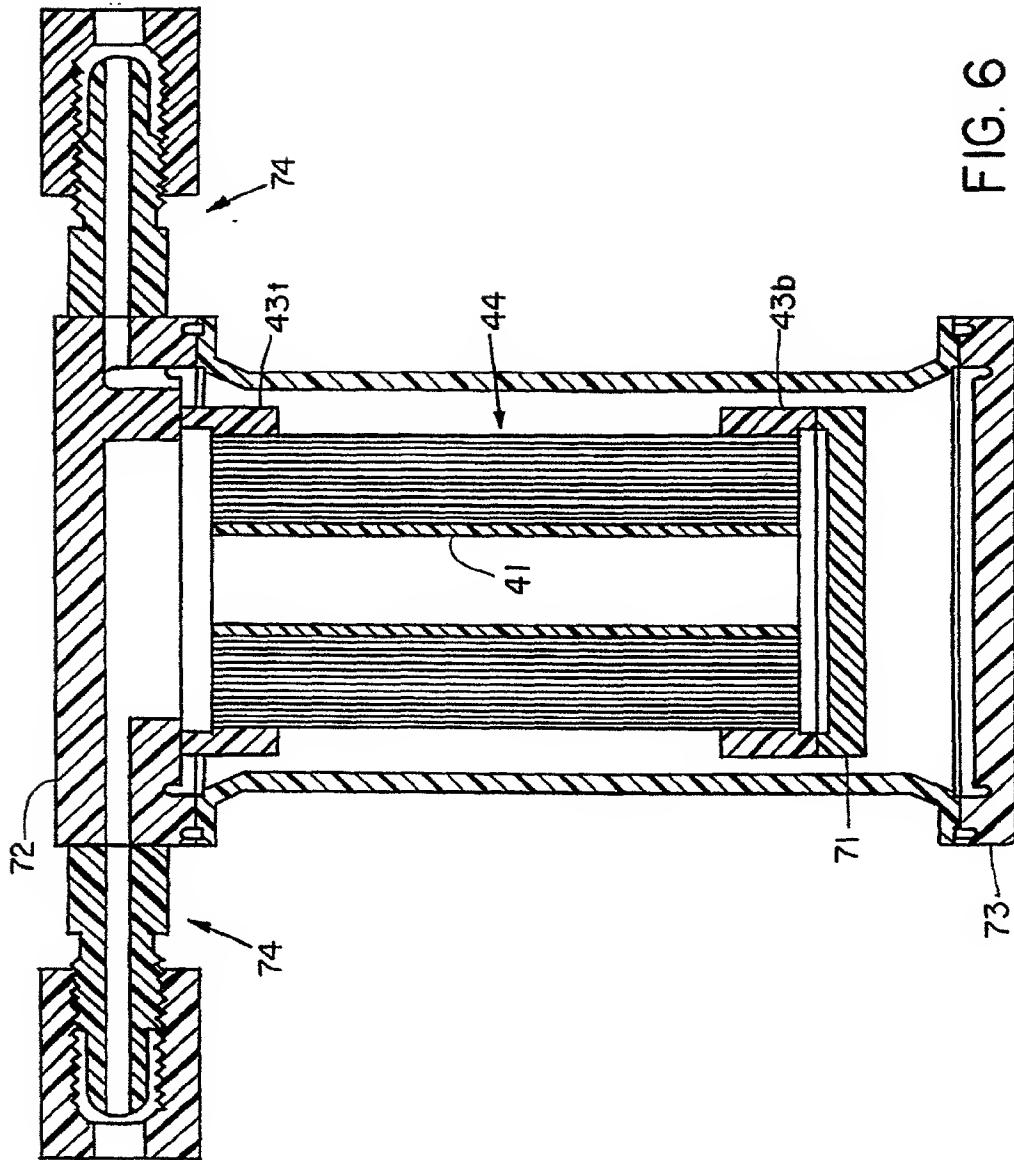


Fig.5



## Declaration and Power of Attorney for Patent Application

### English Language Declaration

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

**"METHOD OF FORMING A PERFLUORINATED, THERMOPLASTIC  
HOLLOW FIBER MODULE"**

the specification of which

(check one)

[ ] is attached hereto.  
[x] was filed on 27 January 2000 as United States Application No. or PCT Application No. PCT/US00/02193  
and was amended on \_\_\_\_\_  
(if applicable)

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose to the United States Patent and Trademark Office all information known to me to be material to patentability as defined in Title 37, Code of Federal Regulations, Section 1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, Section 119(a)-(d) or Section 365(b) of any foreign application(s) for patent or inventor's certificate, or Section 365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate or PCT International application having a filing date before that of the application on which priority is claimed.

Prior Foreign Application(s)

Priority Not Claimed

<u>(Number)</u>	<u>(Country)</u>	<u>(Day/Month/Year Filed)</u>	[ ]
<u>(Number)</u>	<u>(Country)</u>	<u>(Day/Month/Year Filed)</u>	[ ]
<u>(Number)</u>	<u>(Country)</u>	<u>(Day/Month/Year Filed)</u>	[ ]

I hereby claim the benefit under 35 U.S.C. Section 119(e) of any United States provisional application(s) listed below:

60/117,856  
(Application Serial No.)

29 January 1999  
(Filing Date)

(Application Serial No.)

(Filing Date)

(Application Serial No.)

(Filing Date)

I hereby claim the benefit under 35 U.S.C. Section 120 of the United States application(s), or Section 365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of 35 U.S.C. Section 112, I acknowledge the duty to disclose to the United States Patent and Trademark office all information known to me to be material to patentability as defined in Title 37, C.F.C., Section 1.56 which became available between the filing date of the prior application and the national or PCT International filing date of this application:

PCT/US00/02193  
(Application Serial No.)

27 January 2000  
(Filing Date)

Pending  
(Status)  
(patented, pending, abandoned)

(Application Serial No.)

(Filing Date)

(Status)  
(patented, pending, abandoned)

(Application Serial No.)

(Filing Date)

(Status)  
(patented, pending, abandoned)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith. (*list name and registration number*)

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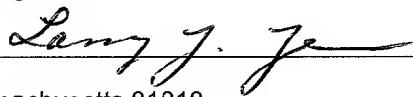
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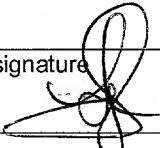
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